

Downstream Details



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Hydrogen Separation Using Inorganic Membranes

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Background

Refiners have found that organic membranes are a reliable and inexpensive way to recover hydrogen from refinery gases. Pressure swing adsorption (PSA) units are also extensively used for this application. Most refineries use high-pressure hydrogenation processes to remove sulfur and reduce aromatic content of products and process feedstocks. About 20% of the hydrogen generated must be purged from these processes to keep hydrocarbons from building up to excessive levels. These purge gases, which may contain 75 volume % hydrogen, are usually used as refinery fuel. Recovery, rather than burning, of this hydrogen for processing petroleum residuum would have signifi-

cant economic benefits to refiners. Thus, a project was designed to explore the applicability of inorganic membranes to separate hydrogen from hydrogen containing hydrocarbon streams. The hydrogen thus separated and purified would be used in the hydrogen processing of petroleum residuum. The value of this process is in the increased yield of light, high value products with a considerable reduction in overall energy usage.

Very high separation factors can be achieved with inorganic gas separation membranes if the pores are small enough to separate gas molecules by molecular sieving. Ideally, a molecular sieve membrane would have pores that are slightly smaller than the larger gas molecule but larger than the smaller gas molecule (**Figure 1**).

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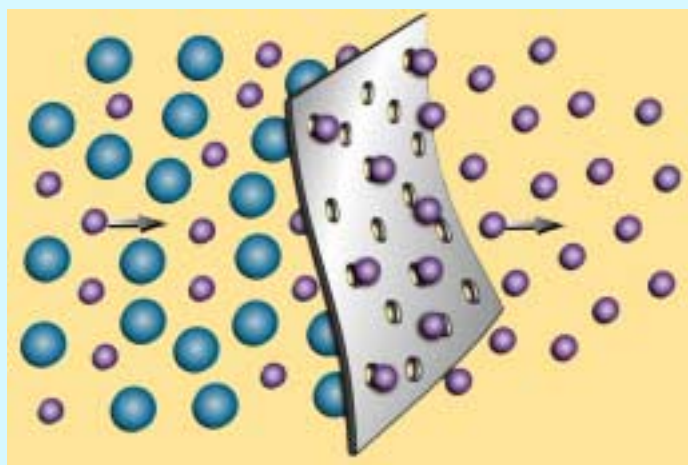


Figure 1: Molecular sieve membrane for separating gas molecules.



Such a membrane permits only the smaller gas molecules (such as hydrogen) to pass through the pores and provides an infinite separation factor. Actual membranes usually have a distribution of pore sizes with some pores larger than ideal. An infinite separation factor is difficult to achieve but is not necessary for economic commercial applications.

It is desirable to have a rugged and robust membrane that can separate and purify hydrogen in gaseous process streams at process stream conditions, without having to cool the gas streams. Significant energy and monetary savings could be realized by a hydrogen separation system that could: (1) operate without cooling the hydrogen containing stream and (2) yield a high quality hydrogen stream containing >90% hydrogen. Polymeric membranes and pressure swing adsorption units are currently used for similar separation. Polymeric membranes are subject to thermal degradation at the high temperatures and pressure swing adsorption requires relatively low-temperature operation and expensive compressors.

Hydrogen Separation Membranes

The objectives of this project are to develop high-temperature (up to 600 °C) membranes that will efficiently separate hydrogen from synthesis gas, refinery purge gases, and hydrogen-carbon dioxide mixtures. To achieve these goals, techniques are being developed for fabricating membranes that have pore diameters of 5 angstroms and that are supported by metal or ceramic substrates.

Development Methods

Several types of inorganic membranes offer potential for hydrogen separation. The inorganic membranes being developed by the Inorganic Membrane Technology Laboratory (IMTL) in the East Tennessee Technology Park (ETTP) include both metal and

ceramic types. These are porous membranes that operate by a molecular diffusion transport mechanism. They are supported membranes, meaning that they are composite structures consisting of a very thin membrane on a porous support structure. The membrane and support may be made of the same or different materials. For example, ceramic hydrogen membranes composed of an alumina membrane on an alumina support are being developed for high temperature (600 °C) synthesis gas applications. For refinery applications, a carbon membrane on a metal (stainless steel) support is being developed. The fabrication methods being employed have been developed at the IMTL. The technology being developed derives from, but is not the same as, technology developed for gas diffusion uranium isotope separation. The hydrogen separation membranes cannot be used to separate uranium isotopes.

To obtain accurate data from test systems, it is important that leak-tight seals be achieved between the ends of the tubular membranes and the test systems. This is particularly important when membranes having smaller pore sizes are tested. A glass glazing technique has been used that provides smooth, impermeable sealing surfaces on the ends of alumina tubes (**Figure 2**).

Although the membrane fabrication processes remain classified for national security reasons, procedures have been established, and approved by DOE, for declaring the membrane products unclassified. This approach assures that if the developments are successful, it will be possible to commercialize these membranes and to use them in unrestricted applications. Pall Corporation, the industrial partner in this project, has made a commitment to pursue this technology with the intent of becoming one of the world's leaders in manufacturing and applications development of ceramic membranes. The ORNL/ETTP/Pall



Figure 2: Smooth impermeable sealing surfaces on the ends of alumina tubes.

Corporation partnership portends the establishment of a strong U.S. position in this important field.

Membrane Testing

This project has made progress toward the development of a working membrane. Pall Corporation has established a manufacturing facility at ETPP.

ORNL will test the membranes produced at the IMTL. The ORNL membrane test system has been constructed and simulated gas streams will be used to test the efficacy of the membranes in making the separations. ORNL is also obtaining a micro test reactor from Phillips Petroleum Company that will be used for testing catalysts. The coupling of the micro test reactor and the ORNL membrane test system will assure that the ORNL and Phillips collaborators obtain meaningful refinery data. It is anticipated that the membrane being developed will be declared unclassified and will be available for testing by third parties in the near future, perhaps as early as the last quarter of FY 2001.

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Downstream Details

Downstream Details is a biannual newsletter devoted to providing information about DOE's Downstream Projects. For more information on these projects, contact **Kathy Stirling**, Project Manager, DOE's National Petroleum Technology Office:

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If you have a project you would like to feature in an upcoming issue, please contact Susan Jackson.

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RTI's TReND Process for Deep Desulfurization of Naphtha

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The U.S. Environmental Protection Agency (EPA) Tier 2 regulations limit the sulfur content of gasoline to 30 ppmw by January 1, 2004. At the same time, the sulfur content of the crude oil in the marketplace is steadily increasing. These divergent trends in sulfur have generated a renewed interest in new and improved desulfurization technologies for fluid catalytic cracked (FCC) naphtha—the chief contributor (>90%) of sulfur in gasoline.

Research Triangle Institute (RTI) is drawing on its expertise in desulfurization of solid fuel-derived syngas (8 issued U.S. patents and a number of pending U.S. and foreign patents) to develop a new process for naphtha desulfurization. RTI has discovered solid regenerable sorbent materials that can selectively remove sulfur compounds, particularly mercaptans, sulfides, disulfides, thiophenes and benzothiophenes, from FCC naphtha vapor without affecting the paraffins, olefins, naphthenes and aromatics (PONA) content.

Regenerable Sorbent Materials

With DOE funding, RTI produced highly attrition-resistant sorbent materials suitable for use in transport reactors. One of these sorbents was produced at commercial scale and was utilized in the Piñon Pine Clean Coal Technology Demonstration Plant for desulfurization of syngas. Using this sorbent expertise, RTI is developing the transport reactor naphtha desulfurization (TReND) process under a cooperative agreement (DE-FC26-01BC15282) with DOE. This process selectively removes sulfur species from naphtha vapor using a solid regenerable sorbent in a transport reactor system. The technology has a number of distinct advantages over the currently available commercial and other emerging technologies.

Transportation Reactor Design

As **Figure 3** shows, the heart of the TReND process is a pair of transport reactors – high velocity, vertical entrained-bed, gas-solid contacting devices. Similar in configuration to that of a fluid catalytic cracker, there are two “risers” – one for desulfurization and one for sorbent regeneration. High velocities (~ 20 to 30 ft/s) manifest into a relatively small footprint for the process equipment. Such a reactor design is only possible because the sorbents used have extremely high attrition resistance. The hydrogen stream reduces the formation of coke during vaporization of the naphtha and assists in the reaction chemistry used to remove the sulfur species. As hydrogen consumption is essentially limited to the desulfurization reactions, hydrogen consumption is small and high hydrogen purity is not required: 50% or greater H₂ concentration will probably suffice.

Ballpark economic analyses suggest that capital and operating costs will be very attractive, compared to conventional hydrotreating. On-stream time and operation schedule can be made to match that of a refinery's FCC unit.

The initial concept for a solid regenerable sorbent for naphtha desulfurization evolved from the syngas desulfurization research being conducted at RTI. Solid sorbents developed for syngas desulfurization are very effective at removing H₂S and COS from syngas streams. However, these are not the typical sulfur species found in naphtha. The majority of the sulfur in naphtha is present as cyclic sulfur compounds, mostly thiophene and its alkyl derivatives, benzothiophene and some mercaptans and disulfides.

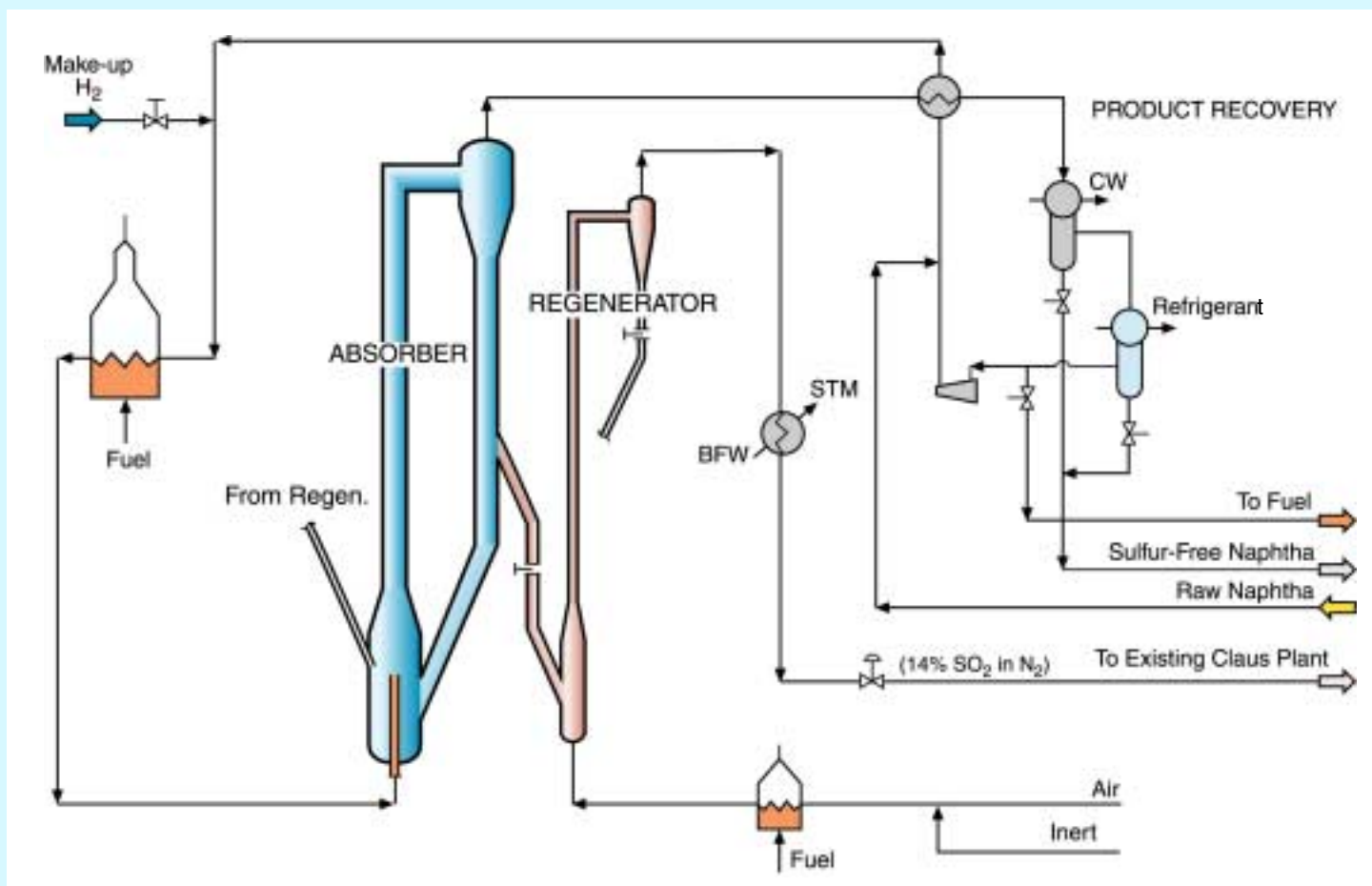


Figure 3: Schematic of the RTI Transport Reactor Naphtha Desulfurization (TReND) process. The heart of the process is a pair of high throughput transport reactors. In the first, naphtha vapor contacts solid sorbent particles for capturing the sulfur compounds. In the second, an air stream regenerates the sorbent and discharges the sulfur as a separate SO₂ stream.

Initial testing at the microreactor and laboratory-scale showed that syngas sorbents, with only minor modifications, could be made to work as naphtha desulfurization sorbents.

Drawing upon these initial positive results, RTI researchers modified the sorbent formulations to enhance their reactivity towards cyclic sulfur compounds such as benzothiophene. Extended multicycle testing, including both desulfurization and regeneration of the sorbent, was conducted to ensure that the desulfurization performance could be maintained. The regeneration of sulfur-laden sorbent was conducted by reacting it with air to produce a sulfur dioxide (SO₂)/nitrogen stream. The SO₂ can then be sent to a claus unit to produce solid sulfur.

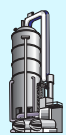
Pilot-Scale High-Pressure Treatment

RTI completed construction of a pilot-scale, high-pressure test unit that is capable of processing naphtha feed at about 1 gal/hr at the commercial conditions of 500 to 800 °F and 150 psig. The initial runs (started in August 2001) will be used to prove out sorbent formulations and determine the optimum operating conditions for larger pilot-plant testing in a 20 gal/hr transport reactor test unit. The next step in the plan to commercialize this promising technology will be teaming with a refinery partner to demonstrate the technology using a slipstream of FCC naphtha in a refinery.

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Development of a Regeneration System for Solid Catalyst Alkylation Using Supercritical Fluid Regeneration

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Alkylation is an important process in modern refining, producing 12% of the gasoline pool. U.S. daily alkylate production exceeds one million barrels with production valued at \$10 billion/year in the U.S. and \$20 billion/year worldwide. Alkylation contributes significantly to the overall economics of gasoline production by converting light hydrocarbons to gasoline range products. In fact, alkylation is in the forefront of the available methodologies for increasing the octane performance of unleaded gasoline. Isoparaffin alkylation is used by the petroleum refining industry to couple high vapor pressure isoparaffins and olefins to yield a low vapor pressure, high-octane gasoline blend stock.

Originally developed in the late 1930s to meet the fuel requirements of high performance aviation engines, alkylation is now primarily used to provide high octane blending components for gasoline. Today's alkylation process removes environmentally undesirable high vapor pressure paraffins and olefins from the gasoline blending pool. Alkylate contains essentially no sulfur, very low levels of benzene, aromatics and alkenes. Alkylate is the cleanest gasoline blending stream produced in a refinery and is an essential component for producing environmentally sound fuels, and is the only existing gasoline component meeting DOE's ultra clean fuel definition.

Current Alkylation Processes Risky

Current industrial alkylation processes are carried out by conducting the reaction in concentrated liquid hydrofluoric (HF) or sulfuric acid (H_2SO_4), both acids are effective catalysts. Both processes are under strong public scrutiny for their potential envi-

ronmental and health affects. HF is especially hazardous, causing serious injury upon contact. Hydrofluoric acid is one of the strongest and most corrosive acids known. Indeed, the toxic nature of hydrofluoric acid has earned it a place on the EPA's "ultra hazardous" chemicals list. A typical alkylation unit is charged with 400,000 lb. of HF, and a catastrophic release of catalyst to the environment could pose an environmental and health problem. Unfortunately, cities have encroached on refineries and now some alkylation plants are located near residential areas. Therefore, extraordinary care must be taken during the use of this acid.

Production of ultra clean fuel is at risk and its growth is limited unless a replacement process can be developed. In order for alkylate to be used as a high volume ultra clean fuel, an alternative safe and environmentally acceptable alkylation process is required. Ultra clean gasoline production by alkylation could be increased by expanding the range of alkenes fed to the alkylation plant, by changing operations at the refinery catalytic cracking units to improve light alkene yields, by increasing the throughput to catalytic cracking units, and by using feedstocks that would have previously been used to make methyl-tert-butyl ether (MTBE). Alkylate's low vapor pressure would allow greater blending of the higher vapor pressure C_5/C_6 isomerate, which would further increase total ultra clean gasoline volume.

Solid Catalyst Development Problematic

Solid acid catalysts could replace liquid acids and eliminate many safety and environmental concerns. However, solid catalysts deactivate rapidly due to depo-

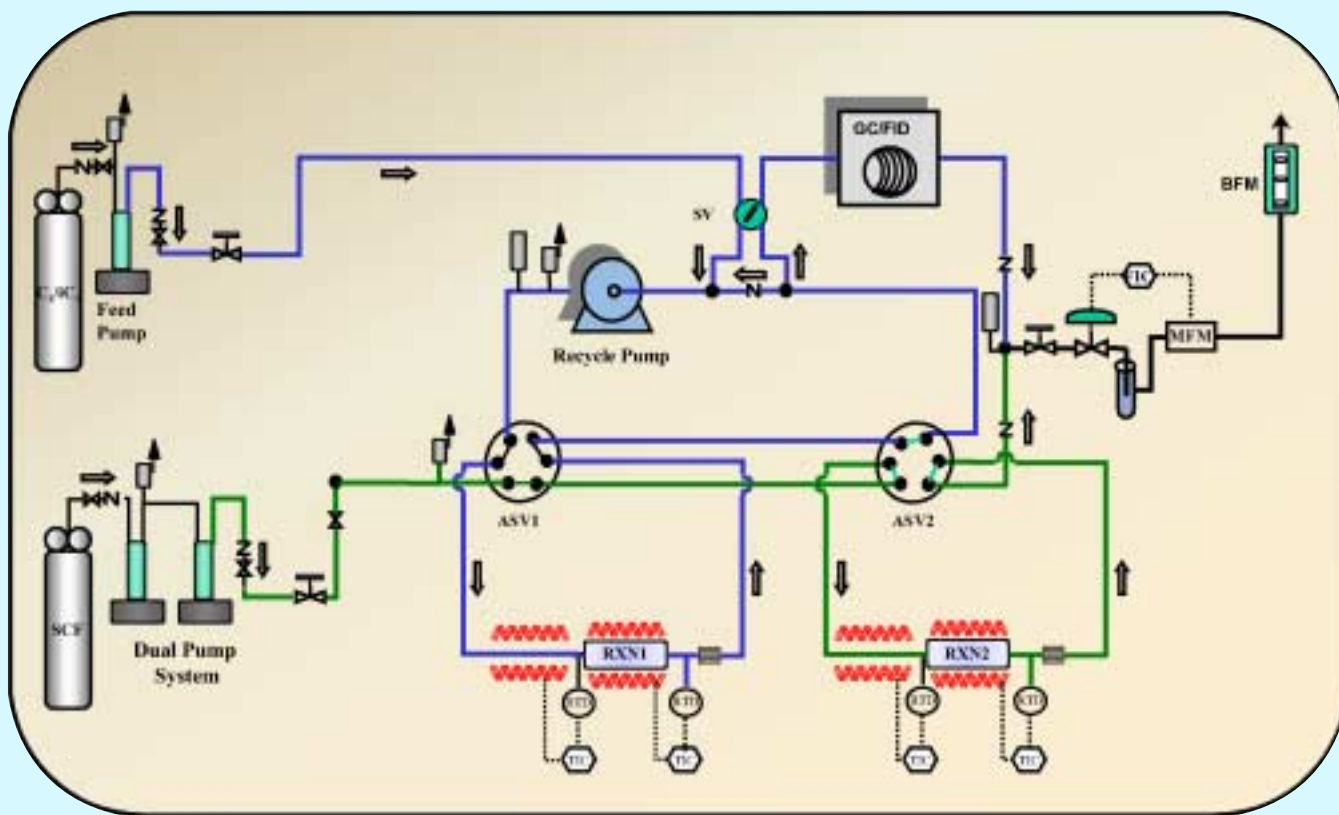


Figure 4: Schematic of the automated experimental system for continuous reaction and regeneration.

sition and buildup of heavy hydrocarbons on the catalyst surface. In addition, solid acid catalysts are difficult to regenerate. Typical oxidative catalyst regeneration processes destroy significant levels of acidic alkylation catalyst activity. Most solid alkylation catalysts can only be oxidatively regenerated 3 to 5 times. This results in high levels of catalyst consumption, making solid catalytic alkylation less economically competitive with H_2SO_4 or HF alkylation. Further, oxidative solid catalyst regeneration results in decreased energy efficiency. A novel, energy efficient regeneration process that can repeatedly regenerate solid-acid alkylation catalysts without significant loss of activity is necessary for the development of an economically viable replacement for the liquid acid catalysts.

Super Critical Fluids

In this project, the INEEL is working to develop an economical alkylation regeneration process using supercritical fluids (SCFs) to regenerate deactivated

solid acid catalyst. INEEL has been exploring SCF/solid catalyst alkylation since 1994, primarily through DOE's National Petroleum Technology Office funding. A completely automated, reaction/regeneration bench-scale experimental system, shown in **Figure 4**, has been developed and tested. An industry team has been assembled that is well positioned to commercialize the technology. The technology team consists of technology development and refining companies that have the ability to evaluate, develop, pilot, demonstrate and license the process to the refining industry.

To date, laboratory bench-scale work has shown that near-critical SCFs are highly effective at regenerating solid alkylation catalyst. INEEL demonstrated that SCF regeneration can provide at least 34 cycles of a single batch of catalyst resulting in greater than 8 days of solid catalyst alkylation while the activity of the catalyst remained greater than 90% of initial activity. ➡

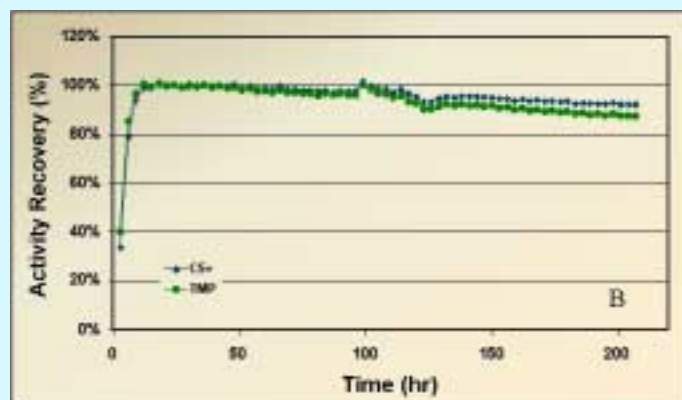
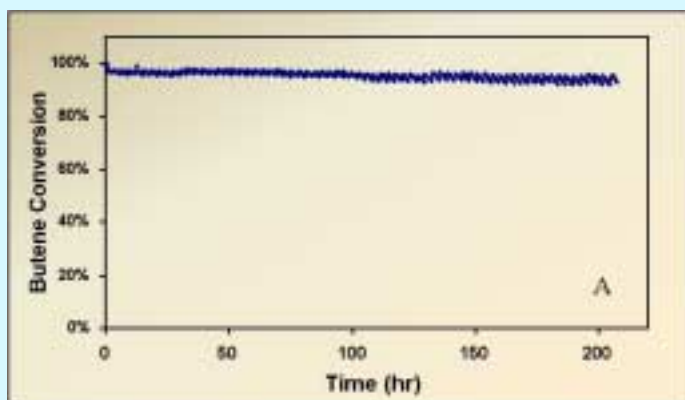


Figure 5: Continuous solid catalyst alkylation reaction/regeneration experiment results showing (A) butene conversion and (B) catalyst activity recovery. Reaction conditions: olefin weight hour space velocity (OWHSV) = 0.2 g olefin/g catalyst/hr, isoparaffin to olefin (iP:o) ratio = 20:1.

Butene conversion and catalyst activity are shown in **Figure 5**. The pilot tests used two reactors where the reactors were alternately switched from reaction to regeneration conditions. Results were obtained at conditions that provided industry-desired space velocities, conversions, yields and selectivities. This technology has resulted in greater than an order of magnitude improvement in product yield per unit of catalyst. Initial experiments using an industrial refinery olefin feed have demonstrated similar results.


Current INEEL efforts examine the reaction/regeneration conditions to determine the maximum number of regenerations, minimum pressure and energy requirements, minimum SCF usage requirements, potential for SCF recycle and reuse, and reactor design requirements for continuous reaction and regeneration.

An Economical, Safe Process

If successful, this project will produce an economical process capable of replacing liquid acids for alkylation. A solid catalyst process would not only eliminate the safety and environmental risks associated with the liquid acids, but also allow for the continued and increased production of high-octane,

ultra-clean gasoline.

The conversion to a solid catalytic process would eliminate: (1) Purchase and transport of 16,000 tons per year of concentrated HF and 2 million tons per year of concentrated H₂SO₄; (2) Storage of approximately 2 million gallons of HF at refineries; and (3) Production and disposal of 200,000 tons per year of hazardous acid-oil sludge. Elimination of these acids would result in savings of approximately \$250 million per year and would significantly reduce safety and environmental risks. The liquid acid alkylation process currently consumes approximately 100 trillion BTUs per year, the equivalent of 20 million bbl of oil. A successful SCF regeneration of solid acid catalyst for alkylation could reduce energy consumption by 50%, conserving 50 trillion BTUs per year. Thus, up to \$350 million in savings could be realized from a SCF solid catalyst alkylation process by cutting energy usage and eliminating liquid acids.

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Petroleum Biorefining for Pollution Prevention

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Background

With the decline in the production of light and medium weight crude oils, refineries are processing heavier and more sour crudes. These crude oils are high in sulfur, nitrogen, and metals. Existing thermochemical processes, such as hydrodesulfurization, can remove much of the sulfur and nitrogen from petroleum but selective removal of all organically bound sulfur and nitrogen, and the removal of metals cannot be accomplished using currently available technologies. The specificity of biochemical reactions far exceeds that of chemical reactions. The selective removal of sulfur from dibenzothiophene by biochemical reactions performed by a mesophilic microorganism has been demonstrated. This technology is now being commercialized for the biorefining of petroleum. Biorefining can also potentially be used to remove nitrogen and metals from petroleum, but so far this area of research has received very little attention.

The focus of this work is to develop new processes using microbial cultures for selective removal of contaminants from petroleum feedstocks without diminishing fuel value. The contaminants - sulfur, nitrogen and metals - contribute to pollution, interfere with refinery operations and corrode equipment. This adds to the cost of refining through increased expenses of regulatory compliance, reduced operating efficiency and diminished product yield.

Microbial Removal of Contaminants

In this project, microbial cultures are being isolated and characterized that can metabolize organic compounds to selectively remove nitrogen, sulfur and metals, from petroleum. In addition to isolating bac-

teria that possess enzymes that selectively cleave C-N or C-S bonds at thermophilic temperatures, genes that encode for these enzymes and then express those genes in the thermophile *Thermus thermophilus* are being identified. The ability to genetically modify and efficiently grow *T. thermophilus* will be an important milestone in the development of bioprocesses capable of operating at thermophilic temperatures. The benefits of a biorefining process that efficiently removes feedstock contaminants are improved refining efficiency; reduced pollutant production; and lower regulatory compliance costs, and possibly lower processing costs.

Bond-Cleaving Bacteria

New microorganisms that can selectively cleave carbon-nitrogen or carbon-sulfur bonds are important because they can be the source of enzymes needed to treat the broad range of organonitrogen and organosulfur chemical structures found in petroleum. Enzymes with broad substrate ranges, high specific activities, and high tolerance to high temperatures are needed. Most crude oils that are in the greatest need of upgrading are viscous and require temperatures of 65 °C or higher in order to be treated as fluids. Accordingly, a biorefining process to treat petroleum would have to operate at thermophilic temperatures and be far more efficient than bioprocesses employed in the pharmaceutical industry. In order to achieve the efficiency needed for a viable petroleum biorefining process it is necessary to express high levels of C-N or C-S bond-cleaving enzymes in large volumes of thermophilic host cells. This requires that the technology to genetically manipulate thermophiles and to efficiently grow thermophiles must be developed and demonstrated. The genetic tools and ➡

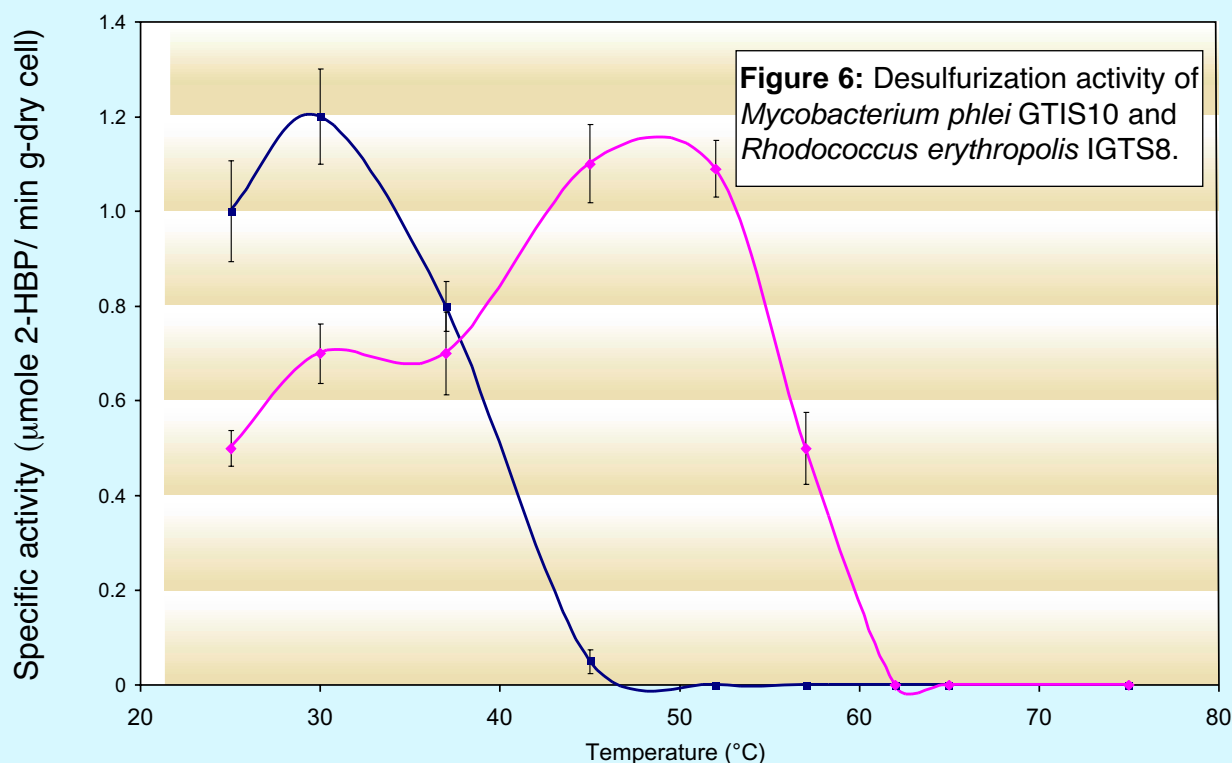


Figure 6: Desulfurization activity of *Mycobacterium phlei* GTIS10 and *Rhodococcus erythropolis* IGTS8.

fermentation data developed in this project for *T. thermophilus* are key accomplishments in the development of a petroleum biorefining process and the development of any bioprocess capable of functioning at thermophilic temperatures.

Mycobacterium Phlei GTS10

An organosulfur-utilizing culture, identified, as *Mycobacterium phlei* GTIS10, was isolated based on its ability to use dibenzothiophene as a sole sulfur source at 50 °C. The desulfurization activity of *Mycobacterium phlei* GTIS10 was compared with that of *Rhodococcus erythropolis* IGTS8 that grows optimally at 30 °C. Interestingly DNA sequence analysis revealed that the desulfurization genes in both cultures are identical yet the temperature ranges over which desulfurization activity can be demonstrated varies dramatically between the two cultures. Analysis of the thermostability of individual desulfurization enzymes indicates that the gene *dszB* is the most thermolabile showing profound inactivation by exposure to temperatures as low as 45 °C for as brief a time as 30 minutes. Nonetheless, the complete desulfurization pathway was demonstrated to func-

tion to some degree in *Rhodococcus erythropolis* IGTS8 at temperatures as high as 45 °C and in *Mycobacterium phlei* GTIS10 at temperatures as high as 57 °C. In each strain optimum activity corresponds to the optimum growth temperature and the maximum activity at which activity can be detected corresponds to the overall thermotolerance of each bacterial culture.


Figure 6 illustrates the ability of this culture to transform dibenzothiophene to 2-hydroxybiphenyl (2-HBP) at various temperatures. Resting cells of *M. phlei* GTIS10 exhibit specific desulfurization activity at higher temperatures than resting cells of *R. erythropolis* IGTS8. The amount of 2-HBP produced by the conversion of DBT by each culture after incubation for 24 hours at various temperatures was quantified by HPLC. Rate of change in 2-HBP concentration was calculated from the linear portion of the curve, generally the first 4 hours of the incubation. Specific desulfurization activity values recorded are averages of three replicate samples from three separate experiments for a total of nine data points. Standard deviation was less than 10 %.

***Pseudomonas Ayucida* IGTN9**

Similarly, microorganisms that can selectively cleave carbon-nitrogen bonds have been isolated. *Pseudomonas ayucida* IGTN9 can selectively cleave C-N bonds in quinoline (Appl. Environ. Microbiol. 66: 688-693, 2000). Additionally, carbazole is representative of organonitrogen compounds in petroleum and genes from several carbazole-degrading cultures are being isolated and characterized.

In recent work a method was developed to conveniently and accurately quantify gene expression levels in *T. thermophilus*. Specifically, the malate dehydrogenase (mdh) gene can be accurately quantified spectrophotometrically and was chosen as the reporter gene for use in quantitative assays of promoter strength/gene expression. The development and use of genetic tools and the mdh gene to quantify gene expression in *T. thermophilus* was demonstrated

and the results were published (J. Bacteriology 183: 1792-1795, 2001, and Letters in Applied Microbiology 32: 412-418, 2001). These tools will be used to identify strong promoters that will subsequently be used to express genes relevant to a biorefining process in *T. thermophilus*. The *dszC* gene which encodes the dibenzothiophene monooxygenase gene was cloned and expressed in *T. thermophilus* demonstrating the ability to clone and express genes relevant to petroleum biorefining. Parallel experiments are also in progress to determine conditions resulting in high yields of *T. thermophilus* cells and enzyme products. Cell yields of *T. thermophilus* have been obtained in batch culture that are higher than any reported in the literature.

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C A L E N D A R **2001**

October 23, 2001

**American Petroleum Institute 2001
Storage Tank Management and Technology
Conference**
Sheraton Crescent Hotel
Phoenix, AZ
Contact: Whitney Schechter
Phone: 202-682-8158

November 8-9, 2001

**National Petrochemical and Refiners
Association**
Lubricants & Waxes Meeting
Omni Houston Hotel
Houston, TX
Contact: Beverly Hamilton
E-mail: Beverly_Hamilton@npradc.org

November 4-9, 2001

**American Institute of Chemical
Engineers**
2001 Annual Meeting
Reno Hilton
Reno, NV
Contact: AIChE Customer Service Phone:
1-800-242-4363 or
212-591-8100

2002

March 17-19, 2002

**National Petrochemical and Refiners
Association**
Annual Meeting
San Antonio, TX
Contact: Tayna Cooper
Phone: 202-457-0480

November 6-8, 2001

American Petroleum Institute
*Introductory Tank Training on Standards 620,
650, 653, AST Emissions, Regulatory
Compliance*
The Pontchatrain
New Orleans, LA
Contact: Veronica Rice
Phone: 202-682-8519

April 22-24, 2002

American Petroleum Institute
Spring Refining Meeting
Hyatt Regency Hotel
Chicago, IL
Contact: Arnetta Smith
E-mail: smitha@api.org
Phone: 202-682-8149

Another Downstream Information source:

The quarterly Newsletter "The Refiner" reports on the National Centre for Upgrading Technology (NCUT) research results in the areas of primary upgrading of Canadian bitumen/heavy oil and secondary upgrading/refining of bitumen-derived streams. NCUT is a research alliance between the Alberta provincial and the Canadian federal governments that performs fundamental and applied research and technology development as well as it provides general research and hydroprocessing catalyst testing services to industrial clients world-wide. For information on how to receive this newsletter contact Dr. Zbigniew Ring by email: zring@nrcan.gc.ca or by telephone at 780-987-8679.



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FALL 2001